C/SIC LIFE PREDICTION FOR PROPULSION APPLICATIONS

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ABSTRACT

Accurate life prediction is critical to successful use of ceramic matrix composites (CMC). The tools to accomplish this are immature and not oriented toward the behavior of carbon fiber reinforced silicon carbide (C/SiC), the primary system of interest for many reusable and single mission launch vehicle propulsion and airframe applications. This paper describes an approach and progress made to satisfy the need to develop an integrated life prediction system that addresses mechanical durability and environmental degradation of C/SiC. Issues such as oxidation, steam and hydrogen effects on material behavior are discussed. Preliminary tests indicate that steam will aggressively remove SiC seal coat and matrix in line with past experience. The kinetics of water vapor reaction with carbon fibers is negligible at 600°C, but comparable to air attack at 1200°C. The mitigating effect of steam observed in fiber oxidation studies has also been observed in stress rupture tests. Detailed microscopy of oxidized specimens is being carried out to develop the oxidation model. Carbon oxidation kinetics are reaction controlled at intermediate temperatures and diffusion controlled athigh temperatures (~1000°C). Activation energies for T-300 and interface pyrolytic carbon were determined as key inputs to the oxidation model. Crack opening as a function of temperature and stress was calculated. Mechanical property tests to develop and verify the probabilistic life model are very encouraging except for residual strength prediction. Gage width is a key variable governing edge oxidation of seal coated specimens. Future efforts will include architectural effects, enhanced coatings, biaxial tests, and LCF. Modeling will need to account for combined effects.

INTRODUCTION

The complex and demanding environments of advanced propulsion systems for future space transportation vehicles involve high temperatures (1650 to 3000°C), low and intermediate temperatures that can also be a problem, high pressures (e.g. to ~6000 psi), severe chemical environments (steam, oxygen and hydrogen under oxygen rich or fuel rich conditions), high gas velocity, exposure cycles from minutes in rockets to hours in some combined cycle approaches, and extremely severe thermal transients and gradients. The application of ceramic matrix composites (CMC) to these systems may provide benefits in terms of life, performance, temperature margin, and weight savings.

For implementation of ceramics to occur for load bearing applications, reliable performance and accurate life prediction are absolutely essential. Current state-of-the-art CMC

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life prediction methodologies embodied in NASALife[1] and similar codes are based on empirical formulations. In general, these models have to be calibrated using experimental data. A shortcoming of these approaches is that changes in fiber architecture, constituent volume ratios, or other variables make the material system completely "new". This requires that the empirical relations be recalibrated by extensive additional experimental testing. Much of this additional cost and time can be reduced if the analytical models are physics based and placed in a micromechanics framework. Once calibrated for a specific CMC system, the predictive capability of the model can then be utilized without additional calibration. Development of this code and similar codes has focused on material systems that are markedly different from the carbon fiber reinforced silicon carbide (C/SiC) composite that is the focus of this study. These codes are lacking because they are not physics-based for accurate prediction of damage due to fatigue and fracture loading conditions. They also do not account for environmental degradation effects due to water vapor attack of silica scales and carbon oxidation that are expected to be major factors in the application of C/SiC to space propulsion systems. Thus, current methods, and the underlying empirical equations upon which they are based, are inadequate for predicting the reusable life of C/SiC space propulsion hardware.

The approach outlined in this paper is designed to resolve these shortcomings. Our objectives are to provide physics based models for the complex interactive mechanical and environmental degradation mechanisms that control C/SiC life, to address mechanical property measurement and prediction from a statistical point of view, and to provide the results as inputs to a parallel micromechanics modeling task.

RESULTS AND DISCUSSION

The overall effort focuses on providing a robust life prediction methodology that will allow confident determination of the reusable life capability of C/SiC space propulsion hardware. This will be accomplished by modifying NASALife to capture the damage and degradation mechanisms associated with static and cyclic thermal and mechanical loading of C/SiC components in a high temperature, high pressure, steam containing environment. Standard C/SiC (1K T-300 fibers, plain weave, pyrolytic carbon interface, SiC matrix and seal coat) from GE Power Systems Composites, LLC was chosen as the baseline material for this study. Enhanced C/SiC, with and without a life enhancing coating, is also being tested.

LIFE MODEL

Physics based, probabilistic lifing models are being pursued. The models will address issues inherently related to composite materials – stochastic characterization of strength, life, and orthotropic material response. Stress rupture tests are currently being carried out in appropriate environments in support of model calibration and validation, and fatigue testing is planned. The lifing models developed will be

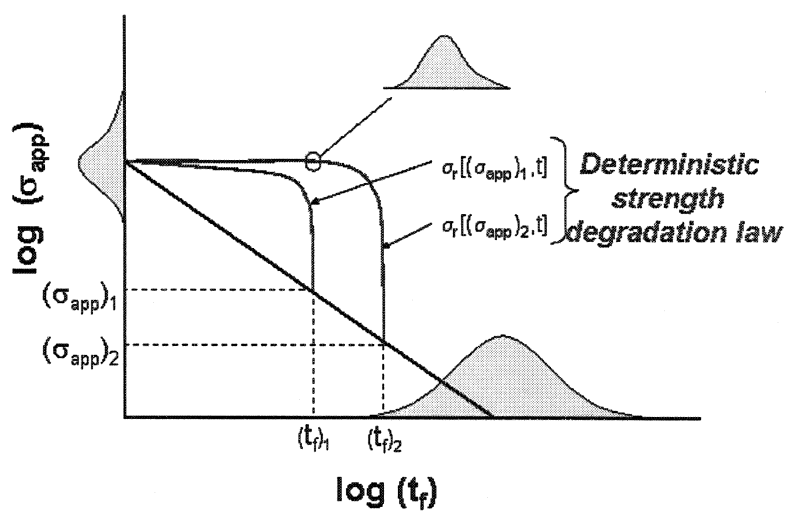

Figure 1. Probabilistic Residual Strength (PRS) modeling approach

implemented in NASALife. A parallel effort for a micro-mechanics (fiber /coating /matrix) based approach to predict stiffness, strength, and life at the coupon level is also being pursued [2, 3, 4]. These on-going tasks have led to a library of computer codes developed specifically for the design of CMC, and they will be adapted to C/SiC to provide state of the art design tools.

Lifing schemes, such as those contained within NASA Life and currently employed for CMC, are adapted from models originally developed for design with metals. These traditional models are comprised of modified Miner's rules, rain-flow calculations, empirical knockdown factors, safety factors, etc. Under this task, a probabilistic residual strength model is being pursued. Residual strength is taken as the damage metric for stress rupture and mechanical fatigue life models. Initial static strength, intermediate residual strength, and time or cycles to failure are all treated as random variables with similar distributions (see Figure 1). In addition, efforts are underway to develop physics based models at the fiber/matrix level for life determination, and environmental effects. In the meantime, the residual strength model utilizes empirical relationships where needed, but is open to modification and incorporation of new models, such as micro-mechanical models and models for environmental degradation, as they become available. Some of the initial results will be shown in the section on Mechanical Testing.

OXIDATION

Oxidation is one key aspect of the environmental attack problem. It arises because C/SiC composites have a microcracked SiC matrix in the as-produced condition. As a result, the pyrolytic carbon coating on the fibers and the carbon fibers themselves are subject to oxidation attack when the cracks are open[5, 6]. This degradation mechanism occurs at temperatures below the composite fabrication temperature under zero stress conditions, and at all elevated temperatures sufficient for oxidation of the fibers (>400^{o}C) when stress sufficient to open matrix cracks is applied. Figure 2 illustrates the role of thermal expansion mismatch between the SiC matrix and the C fibers, and of applied stress on crack opening. Since oxidizing conditions are expected to be present in the service environment of most C/SiC components, prediction of oxidation attack is a key ingredient of the life prediction model. A more thorough understanding of the effects of environment, temperature, and stress on the degradation of carbon fibers is being developed so that material limitations can be better identified and methods of improving oxidation resistance can be addressed. The development of a physics and experimentally based fiber oxidation model is being pursued as depicted in Figure 3. It incorporates such variables as reaction rate, diffusion coefficient, temperature, partial pressure, and environment. It tracks the recession of an array of fibers in a cracked matrix so that the oxidation kinetics involved in carbon fiber degradation can be studied. Oxidation studies, stress rupture tests, and microscopy are being conducted to aid in the

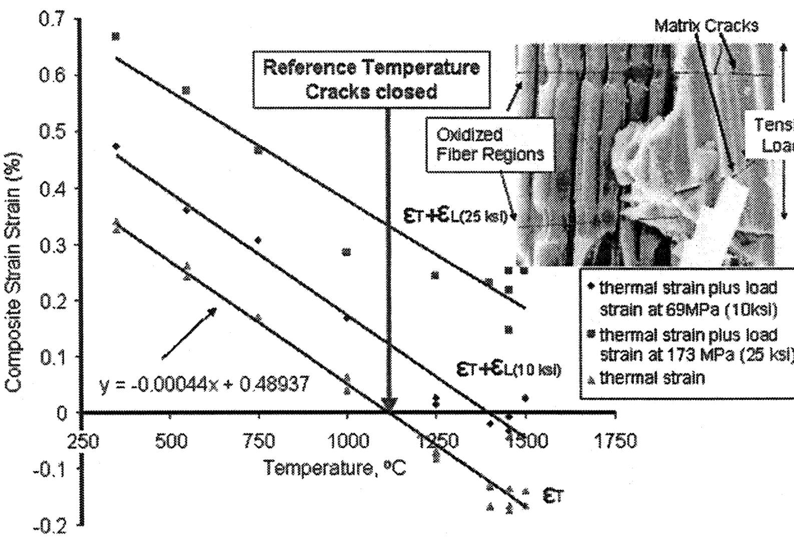

Figure 2. Crack opening determined by load and thermal strain

development of the model. Figure 4 illustrates how a small amount of oxidation attack of T-300 fibers within a composite exposed at intermediate temperatures in air seriously degrades tensile strength. Fibers throughout the composite are damaged. At higher temperatures where tow inner fibers and inner tows are shielded from attack the mechanisms of fracture are different. An outer damaged or consumed case leads to overload of a relatively undamaged core.

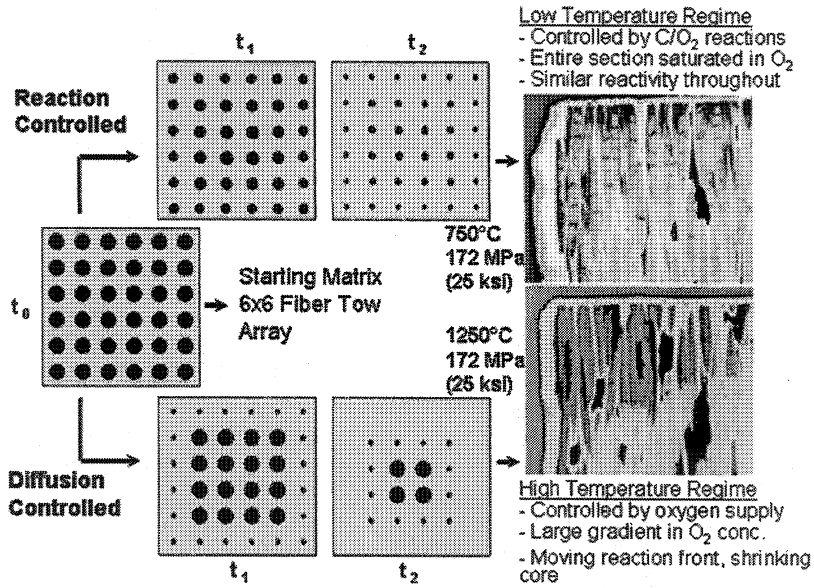

Figure 3. Carbon fiber attack mechanisms are temperature dependent

MECHANICAL TESTING

The test plan for tensile, creep-rupture, and fatigue testing was formulated to satisfy several requirements: (1) Calibration and verification of the probabilistic residual strength (PRS) model, (2) assessment of usable service life for various conditions (i.e. temperature, stress, and environment) for C/SiC, and (3) determination of the effect of alternative fiber architecture on material behavior and model capability.

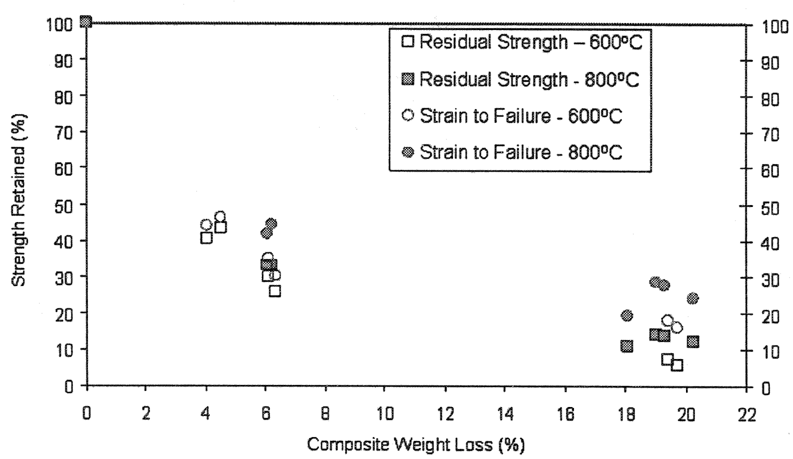

Figure 4. Residual composite tensile strength versus weight loss due to oxidation

The performance of standard C/SiC and enhanced C/SiC in air is shown in Figure 5 as a function of temperature. The enhanced C/SiC has a matrix that is modified with B_4C particulates to allow low temperature glass formation and crack sealing. Enhanced C/SiC outperforms the standard C/SiC at 800 and 1200°C. Tests at other temperatures are planned.

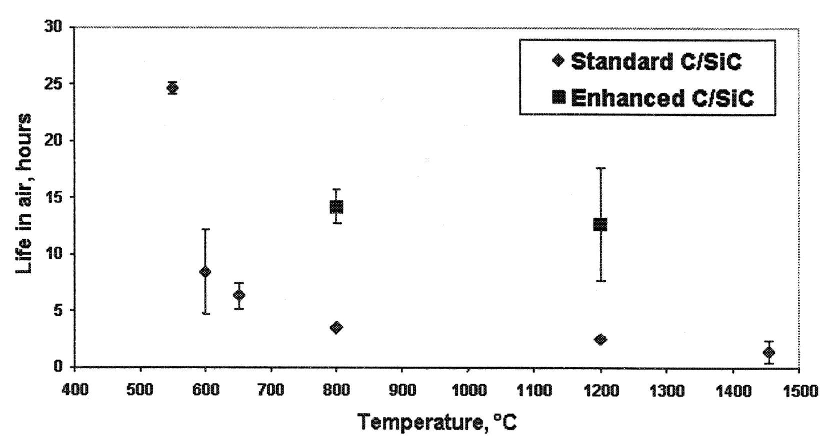

Figure 5. Stress rupture Life for C/SiC as a function of temperature (all tests conducted in air at 69 MPa (10 ksi)

Lives of standard C/SiC

in air are too short for model development data. Thus an artificial condition (1000 ppm O_2 in argon) was selected to give more readily measured life distributions. One study conducted under this effort examined the effect of specimen width on stress rupture life. Data are shown in Figure 6 for tests at 800 and 1200°C. The fact that life increases dramatically with increasing specimen width is encouraging and indicates that component life will likely be significantly longer than predicted by test data generated on the narrow specimens. Attack is much more severe at machined and seal-coated edges and corners than at normally processed surfaces. The rate of attack at edges is about three times the rate on large surfaces. However, since there is far more surface area than edge area, the highest volume of carbon fiber consumption occurs from the large flat surfaces, and in a large panel edge damage would become insignificant from a structural standpoint, as shown in Figure 7. However, performance integrity and sealing between panels would be compromised.

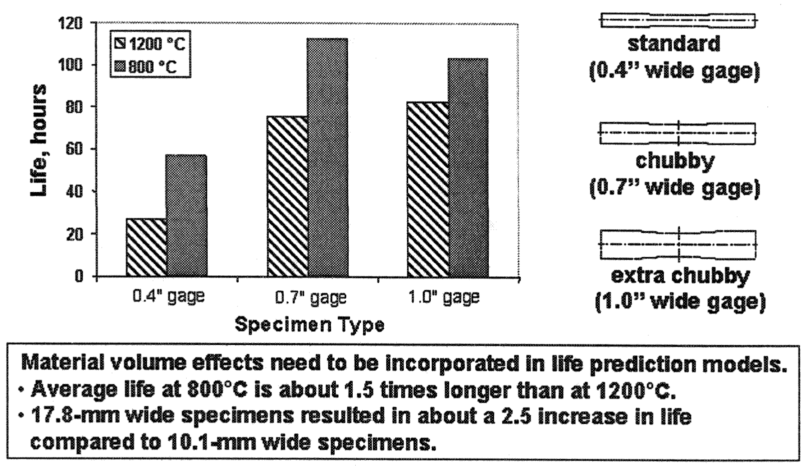

Figure 6. Stress-rupture lives as a function of specimen width for C/SiC (207 MPa (30 ksi), 1000 ppm O_2/Ar)

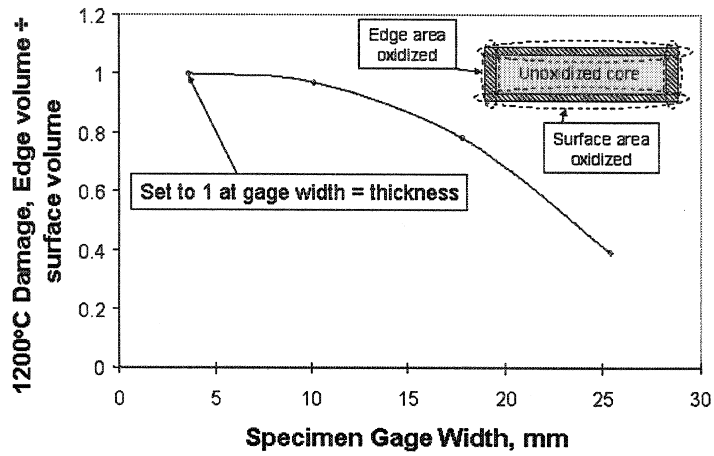

Figure 7. Edge damage effects insignificant for large panels

To develop the PRS model, calibration tests were run at 800 and 1200°C in 1000 ppm O_2 in argon. To verify the model a series of stress rupture tests were run at 207 Mpa (30 ksi). Results at 1200°C are plotted along with two model predictions of probability of failure in Figure 8. At the critical low probability of failure tail of the distribution for both the Kachonov and power law the predictions are in good agreement with the data. Residual strength predictions are shown in Figure 9.

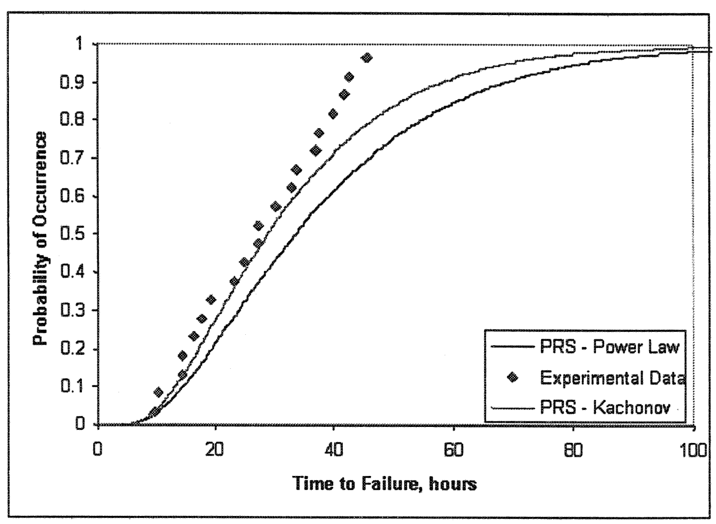

Figure 8. The probabilistic residual strength (PRS) life prediction model predicts failure time behavior at 1200°C, 207 MPa (30 ksi)

The measured median residual

strength is about 240 Mpa (35 ksi) after 15 hours of stress rupture exposure at 207 Mpa (30 ksi), at 1200°C. The Kachonov model predicts a much more gradual strength reduction with the median strength not far below the as-received strength. The situation is similar after 7.5 hours. Results at 800°C are plotted along with two model predictions of probability of failure in Figure 10. At the critical low probability of failure tail of the distribution for the Kachonov prediction is in good agreement with the data. Residual strength predictions for 800°C are shown in Figure11. The measured median residual strength is about 380 MPa (55 ksi) after 15 hours of stress rupture exposure at 207 MPa (30 ksi), about 138 MPA (20 ksi) higher than at 1200°C. The Kachonov model again predicts a much more gradual strength reduction with the median strength not far below the as-received strength. The power law prediction that fit the data at 1200°C now over predicts damage.

Stress rupture tests to determine the effect of moisture on C/SiC performance were carried out. At 600°C 20% steam in argon is benign in comparison to air. At 1200°C steam is about as aggressive as air. These results can be understood from the fiber oxidation data. At 1200°C fiber oxidation in air and in 20% H_2O in Ar is about the same. At 600°C oxidation of T-300 still occurs rapidly in air, but in 20% H_2O in Ar the reaction kinetics are limiting the reaction rate to near zero.

WATER VAPOR ATTACK

The reaction of silica scales with water vapor is the most straightforward aspect of the environmental attack problem to characterize and model because stress state interactions are insignificant. Current state of the art consists of both experimental data and a model for SiC and Si_3N_4 recession due to formation of

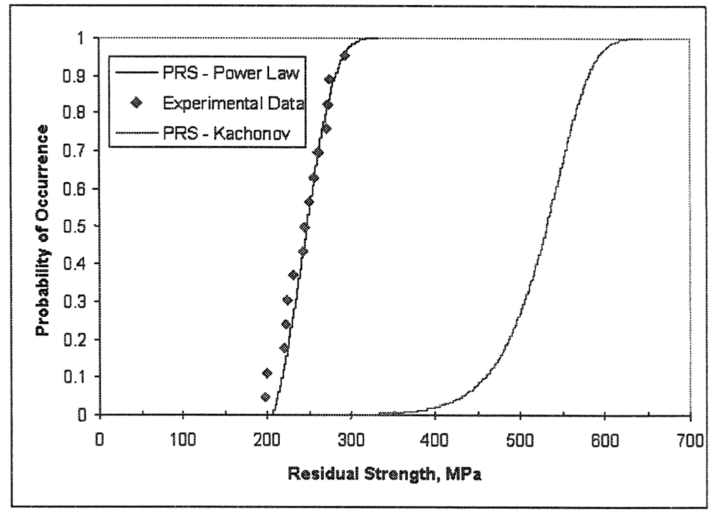

Figure 9. The PRS life prediction model predicts residual strength behavior at 1200°C after 207 MPa (30 ksi), 15 h exposures and RT tensile test.

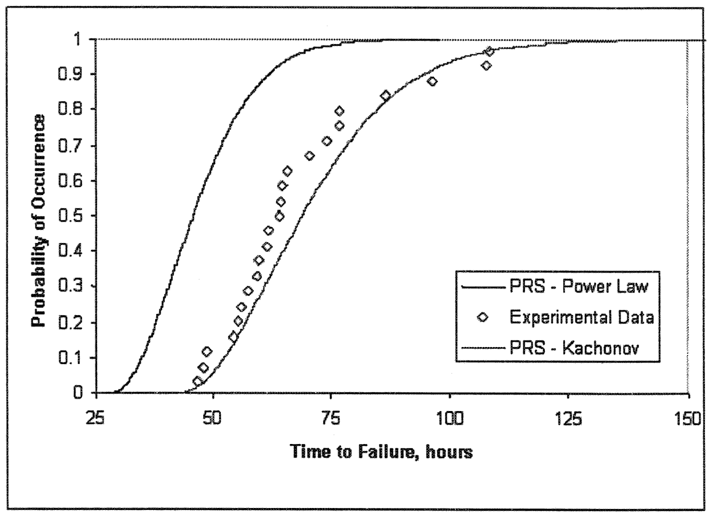

Figure 10. The PRS life prediction model predicts failure time behavior at 800°C, 207 MPa (30 ksi)

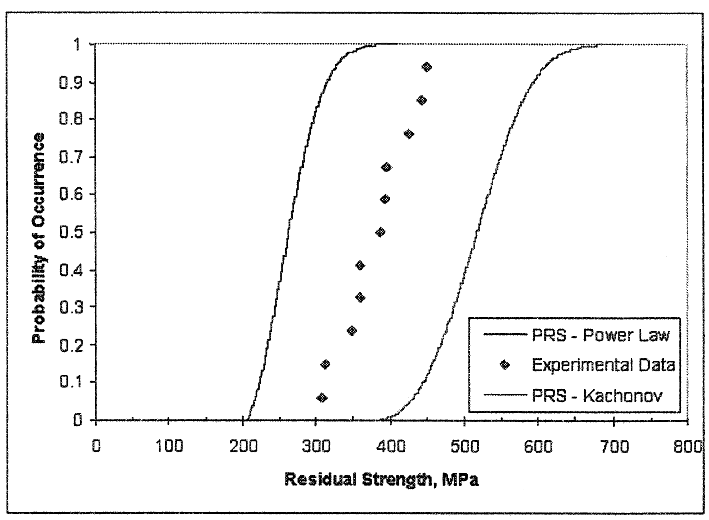

Figure 11. The PRS life prediction model does not fit residual strength data at 800°C after 207 MPa (30 ksi), 15 h exposure and tensile test

volatile silicon hydroxides in combustion conditions typical of aircraft engine[7]. The model predicts material recession rates as a function of water vapor partial pressure, total pressure, gas velocity, and material temperature. In this task, the model is being extended to pressures, gas chemistries, gas velocities, and material temperatures typical of the rocket engine environment. High pressure, high velocity tests were run at various O_2/H_2 mixture ratios. The experimental set up is shown in Figure 12. C/SiC specimens were coated with a 25 mil extra-

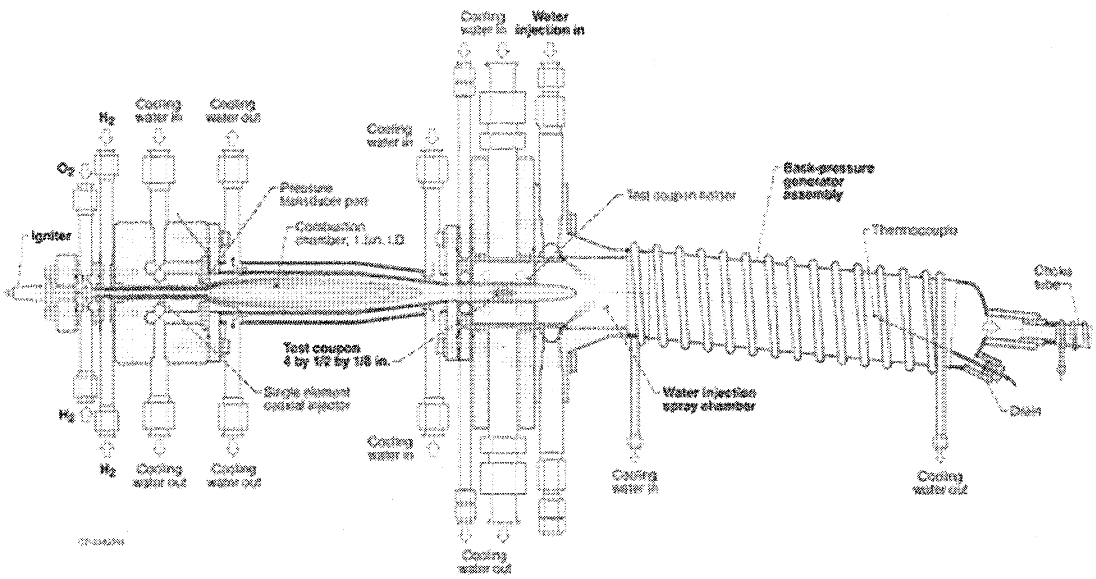

Figure 12. Subsonic high-pressure coupon test configuration used for determination of SiC recession due to moisture generated by combustion of H_2 and O_2

thick SiC seal coat to enable recession measurements to be made. The appearance of three specimens after exposure to three OF (oxidizer to fuel) ratios is shown in figure 13. Damage increases dramatically as temperature increases. Thickness recession data are plotted in Figure 14. At the most severe condition a significant amount of recession has occurred in only 10 minutes of exposure. Recession measurements will be refined by metallographic measurements and results will be compared with model predictions.

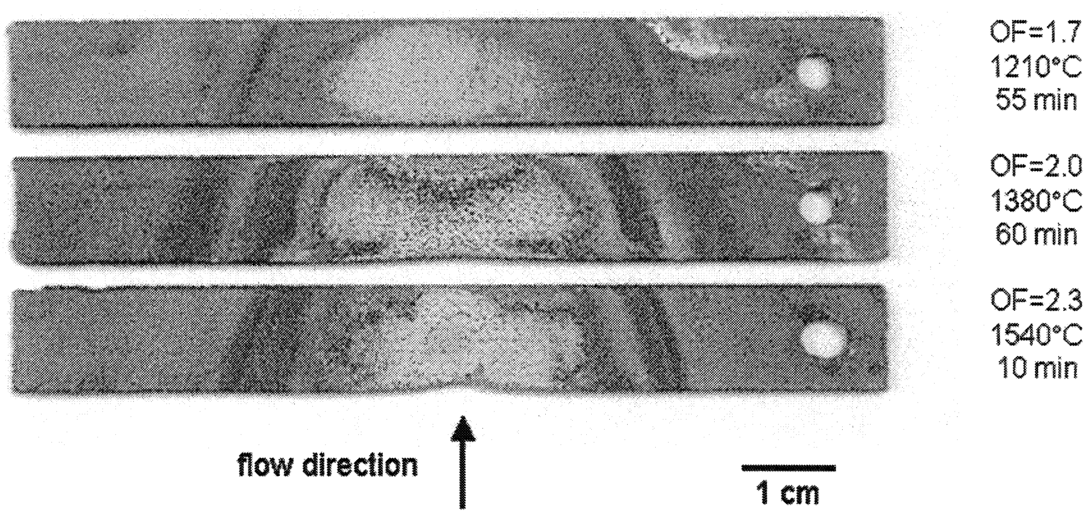

Figure 13. SiC coated C/SiC after exposure to products of H_2 / O_2 combustion

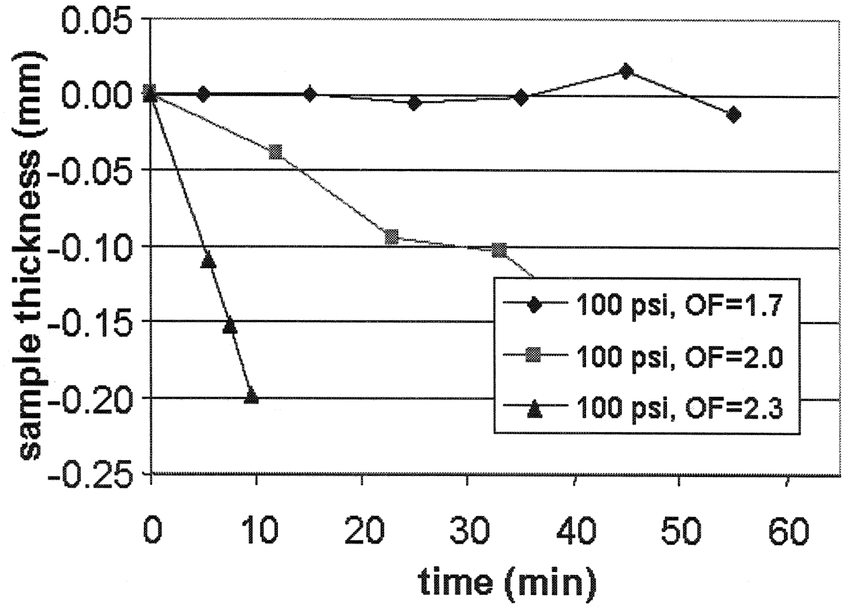

Figure 14. SiC coated C/SiC recession measured as thickness at leadiing edge after specimen exposure to products of H_2 / O_2 combustion

SUMMARY AND CONCLUSIONS

Life prediction for C/SiC is a complex problem involving many interactive mechanisms. The plan outlined here will analyze mechanisms in isolation as well as their interactions, develop mechanistic lifing models, and develop understanding of the importance of statistics in C/SiC behavior.

Progress has been made in all aspects of the plan:

- Steam effects: Preliminary tests indicate that we will see an effect consistent with past experience. The kinetics of water vapor reaction with carbon fibers is slow at 600°C, but comparable to air attack at 1200°C.
- Oxidation model: Tensile strength degradation resulting from low levels of oxidation of the T-300 fibers at intermediate temperatures where cracks are open has been determined to be severe. Ultimate tensile strain degrades in parallel with strength.
- Mechanical property tests and life model: Initial results are very encouraging, but no single version of the model is predicting life and residual strength at all temperatures. This underscores the need for a good physics based model. Edge oxidation of seal coated specimens has been investigated by testing specimens of varying gage width. The mitigating effect of steam observed in fiber oxidation studies has also been observed in stress rupture tests.

FUTURE WORK

Future efforts will include architectural effects, enhanced coatings, biaxial tests, and low cycle fatigue. Modeling will need to account for combined effects and provide a tie in to the

PRS model. The measurement of effects of water vapor in the combustion environment will be carried out and higher pressures.

ACKNOWLEDGEMENTS

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